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Research Paper

Investigation on the different stirring medium on the microstructural and mechanical properties of Aluminum-Titanium Di Boride *in-situ* metal matrix composites

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Abstract

A thorough investigation on the influence on the different stirring medium on the composite melt was carried out in an attempt to prepare In-situ Al1050-5wt.% TiB_2 composite. In the preparation of in-situ composites stirring medium has its own effect on the final prepared composite. The stirring of the melt slurry was made intermittently at regular intervals. The melt was stirring at regular interval was carried out using different stirring medium. The first part of experiment was carried out without stirring of the melt, in the second part the melt was stirred with motor and in the third part of the experiment the composite melt was stirred with manually and Although different stirring medium was used, the intermittent stirring of the melt was kept at constant interval time of 10 minutes.

No uniform formation of particles were found in the composite processed without stirring, when the composite melt was stirred with motor kept at 100 rpm the results showed that the particles were formed, however the particles were not only of TiB_2, there were also presence of other particles such as Al_3Ti and AlB_2. However stirring of the melt manually showed excellent results. The composite processed with manual stirring confirmed the presence of only TiB_2 particle in the melt which confirmed that the reaction was complete.

Due to lack of understanding of the process parameters, partial reactivity of the salts in the alloy matrix occurs, resulting in the development of intermediate phases such as

$AlTi_3$ in the composite. The inclusion of intermediary phases in composites is proven to be detrimental to their characteristics. People have been attempting to manufacture in-situ Al/ TiB_2 composites without the formation of intermediate reaction products since FAS developed in-situ Al/ TiB_2 composites, but only a very few have succeeded.

By choosing proper stirring medium composites with only TiB_2 particles can be prepared. By exactly choosing the stirring medium condition we were able to successfully produce TiB_2 reinforcements in the melt, there by effectively producing Al1050-5%TiB_2 *in-situ* composite. The microstructure of the composites was examined and the mechanical properties were studied. The studies on the microstructural and mechanical properties of the composite produced using manual stirring showed superior properties.

Keywords: XRD, SEM DTA-TGA, ALLOY, Mixed metal composites

INTRODUCTION

Metal matrix composites were developed in response to the increasing mandate for weightless and tougher materials. When compared to monolithic alloys, MMCs were created by adding the beneficial properties of certain metals and specific ceramics reinforcements, resulting in higher strength-to-cost ratios and strength-to-weight. Among metal matrix composites, though the continuous fiber reinforced composites have the effective strengthening competences, their applications have been limited due to difficult production techniques, anisotropy, and the higher cost of continuous fibers. The creation of particle reinforced MMCs was prompted by the high production cost, several health risks due to whiskers, and additionally the dispersion issues seen in short fibers among discontinuously dispersed reinforcements in MMCs. Furthermore, particle reinforced MMCs have found uses in the defence, automotive, aerospace and space industries due to the readiness of a wide range of materials for matrix, low-cost reinforcements, and simple production procedures. The interfacial connection between the matrix and reinforcements must be strong for the load transfer mechanism to work properly. Ex-situ treated composites have inadequate interfacial bonding strength, according to investigations [1]. Furthermore, problems with ex-situ processed composites such as the wettability is very poor, the creation of interfacial products which are undesirable between the reinforcement

and the matrix, and limitation in the reinforcements size of the led to the development of in-situ metal matrix composites. Plethoras of processing processes have been created in response to the realised promise of in-situ metal matrix composites. The processing methods were divided into four categorized into, vapour-liquid solid (VLS), liquid-liquid solid-liquid and solid-solid reaction process [2]. aluminium based have long as the matrix due to their superior qualities such as high specific strength, lower density, excellent castability, strong corrosion resistance, good weldability, and high thermal stability. Aluminium alloys are quite affordable when compared to other metals such as Mg and Ti. Borides, carbides, oxides, and nitrides are common ceramic reinforcements in aluminum-based in-situ composites. Low density of 3.4 g/cm^3 and high Young's modulus of 216 giga Pascal among the available in-situ treated reinforcements [3]. FAS process evolved from a well-known technique for producing master alloys such as Al-Ti-B, which were employed in refinement of grains in aluminium and related alloys [4]. In-situ Al/ Al_3Ti composites are commonly made using the FAS technique by adding Ti-bearing fluoride salts like potassium hexafluortitanate (K_2TiF_6) into the melt of aluminium, where reinforcements such as Al_3Ti are nucleated and grown by the separation of Ti which is existing in the halide salts via a chemical reaction which is exothermic of aluminium and the halide salts added [5]. inclusion of intermediary phases in composites is proven to be detrimental to their characteristics. People have been attempting to manufacture in-situ Al/ TiB_2 composites without the generation of intermediate reaction products since FAS developed in-situ Al/ TiB_2 composites, but only a few have succeeded [6,7]. Titanium di boride is a hard ceramic substance that belongs to the transition metal diboride clan. TiB_2 is a synthetic compound that does not exist in nature. Some of the procedures now used to make TiB_2 powders include fused-salt electrolysis [8], mechanical alloying [9], carbothermal reduction [10], and mechano-chemical synthesis [11]. The Ti atoms are found to be on the unit cells origin, while the other two atoms of B are at the (2/3, 1/3, 1/2) and (1/3, 2/3, 1/2) locations respectively, in the TiB_2 crystal structure [12, 13].

EXPERIMENTAL

Materials

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Al 1050 aluminium alloy, potassium borofluoride, potassium titanium fluoride, hexachloroethane tablets and Coverall 11 for composite fabrication were utilised in this study. Hi-Tech, Mangalore provided the Al1050 aluminium alloy, which was selected and employed as the material for the matrix. An Optical Emission Spectrometer (OES) was used to examine the alloy's composition (Model LMS 04 of spectromax, Germany). Table 4.1 shows the constituent composition of the alloy obtained. From the Madras Fluorine Pvt. Ltd company commercial grade of potassium borofluoride and potassium titanium fluoride in powder form (70 m) were purchased from., Chennai, for use in this study. XRD was used to examine the halide fluxes K_2TiF_6 and KBF_4. Servel provided Coverall 11 and hexachloroethane (C_2Cl_6) pills.

Methods

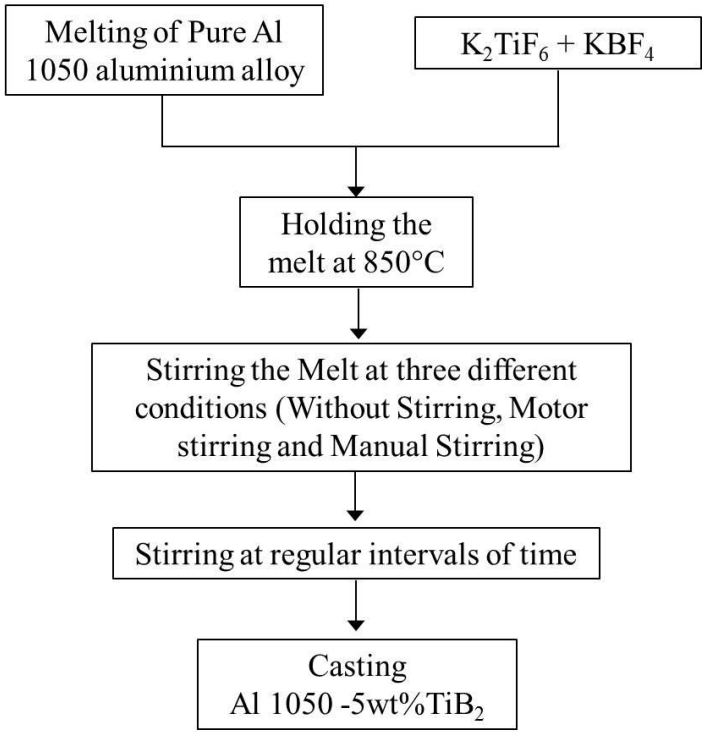

Fig-1 preparation for aluminum Titanium borate

The studies were carried out in order to determine the best processing conditions for Al-5TiB_2 composites. In an electrical resistance furnace heated to the required temperature, the aluminium alloy Al was melted in a graphite crucible. Using a thermocouple which is a K-Type, the temperature of the melt was closely monitored. Protection of the melt was carried

out by using Coverall 11 from contamination the melting process. The melt was degassed by plunging some hexachloroethane by using a graphite plunger as soon as the aluminium melt reached the fixed holding temperature. The halide salt which were packed in the aluminium foil were dried salt and added in batches to the aluminium alloy melt as soon as the melt reaches the set temperature again, and the melt has been intermittently agitated with a zirconia which has been coated on mild steel rod for uniform mixing of the melt. The whole melt was kept at 850°C temperature for the period of one hour after the exactly weighed stoichiometry of the salts were added to guarantee complete the halide salts and the melt. The aluminium alloy melt has been stirred intermittently at regular intervals to achieve uniform dispersion of the precipitated particles and to spark the interaction between the salts and melt. TiB_2 particles were studied in situ. The slag has been decanted from the aluminium alloy composite melt as soon as the reaction completes. The melt was transferred into a 30 mm dia and 170 mm height mild steel mold that had been warmed to around 250°C with the removal of the slag.

RESULTS AND DISCUSSION

X ray-Diffraction analysis

The XRD pattern of the as-cast Al 1050 alloy is shown in Fig. 4.1. When compared to JCPDS data, all of the obtained peaks corresponded to alpha Al which was in exact math with the peaks indicated in the card number 04-0787. There were no other peaks that corresponded to intermetallic other than aluminium. The intermetallic present in the aluminium were not prominently visible. This could be owing to the existence of very low intermetallic of volume percent's, which were substantially below the XRD detection limit. From the figure it is evident that there are five prominent peaks. Among the prominent peaks the plane with (111) is the highest peak with the highest intensity.

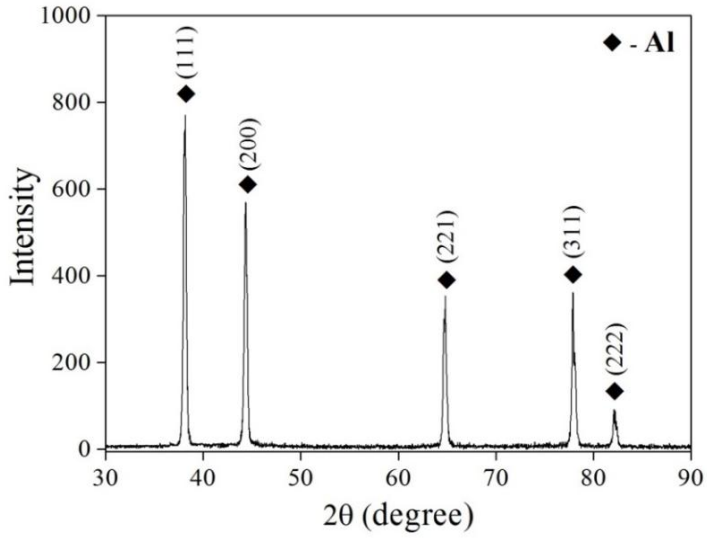

Fig. 2. XRD pattern of alpha-aluminium in the as casted Al 1050

DTA-TGA analysis

Fig.4.10 shows the curve obtained from the TG and DTA analysis. By using this analysis we can find the decomposition behaviour of the added salts to the aluminium melt. The curve was obtained for the temperature range of 30 to 900 ºC.

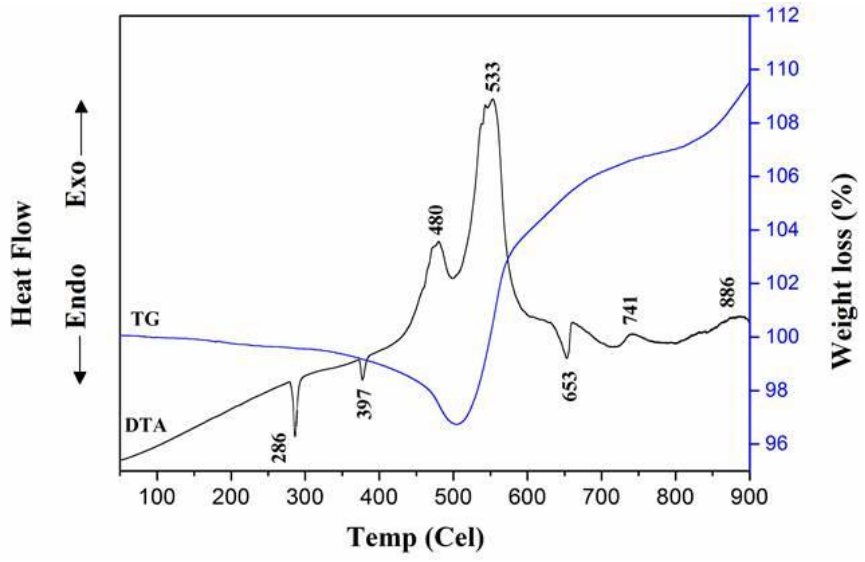

Fig. 3 TG and DTA graph showing K_2TiF_6 and KBF_4 salts with aluminium melt

As the temperature kept on increasing both endothermic and exothermic reaction was evident. The endothermic and exothermic reaction was due to the decomposition reaction. In the DTA curve the first endothermic peak at 286ºC. This peak occurred du the transformation of potassium tetrafluoro borates polymorphic transformation. The second peak at 397ºC which is endothermic is caused by the moisture removal from the fluoride salts. The sample had showed a weight loss of 1.2 percent till the second endothermic reaction. The loss of moisture in the sample had caused the weight loss. Two exothermic peaks at 480 ºC and 533ºC was seen when the sample was further heated.

The peak which is seen at 480ºC was due to the exothermic reaction of potassium tetra fluoroborate with Al. Similarly the peak at 533ºC which is exothermic was due to reaction of potassium hexafluoro borate with Al.

We can see in the TG curve that a weight loss of 2.5% occurred. When the salts started to react with Al by an exothermic reaction some gases such as TiF_4 and BF_3 evolved and which would be the cause for the reduction in weight of the salts. However as the exothermic reaction started the increase in weight of the sample was due to the formation of AlB_2 and Al_3Ti particles. Furthermore, the increase in weight was due to the formation of slag.

Scanning Electron Microscopy

The temperature of the melt has a huge influence on the preparation of the composite. The above photomicrograph obtained using SEM were from the prepared composite at different temperatures. The sample processed without stirring is shown in Fig.4a clearly shows that the particles have formed during the process. The formed particles were of different size and morphology. The string like particles which are seen the photomicrograph was found to be having the elemental signature of Ti, Al and Si. The energy dispersive spectrum confirmed that some of the particles were TiB_2 and Al_3Ti. The micrograph also shows the presence of slag. This was further confirmed by XRD. It can also be observed that without stirring of salts the mixing of salts with the melt is complete. Hence the slag remained in the composite.

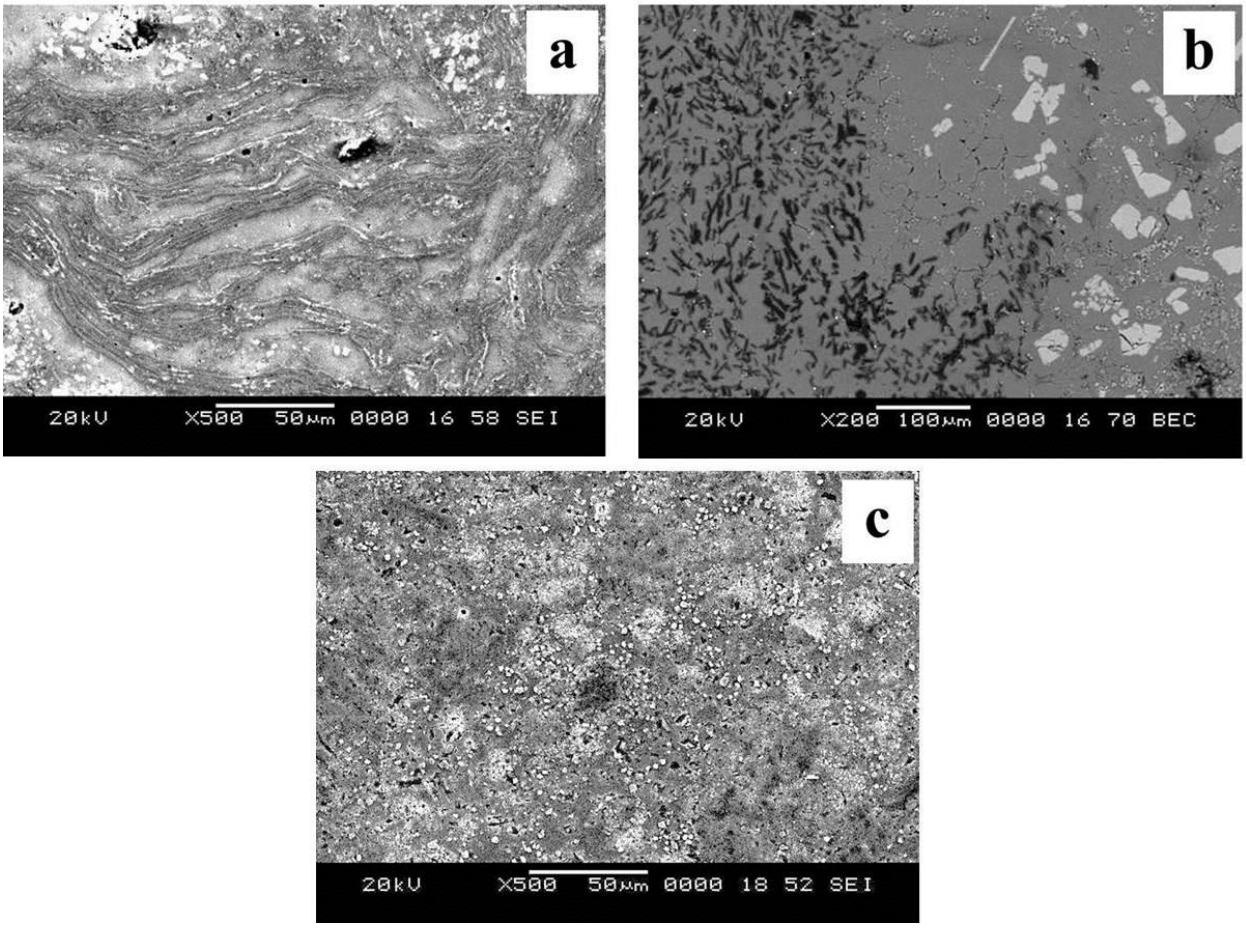

Fig.4 (a-c) Photomicrograph obtained using SEM, from the prepared composite at different stirring medium (a) Without stirring, (b) Motor stirring, and (c) Manual Stirring.

The SEM image of composite processed with manual stirring is shown in Fig.4c. It can also be observed that with manual stirring of salts the mixing of salts the salts with the melt is complete. Hence the slag remained in the composite. Analysis on the image shows that there is formation of only TiB_2 particles. The sizes of the TiB_2 particles were found to be in the range of nano meter to less than 1 µm. The TiB_2 does not form directly from the exothermic reaction of the salts with the melt. First as the exothermic reaction between the salts and melt occur the Ti and B is released from the salts. The Ti and B react with aluminium and form Al_3Ti and AlB_2. As the melt temperature is held for a consistent amount of time, the Al_3Ti and AlB_2 further decomposes and forms as TiB_2.

The EDS analysis on the particles confirmed that they were all Al_3Ti, K_3AlF_6 slag. The SEM image of composite processed with motor stirring is shown in Fig.4b the observation on the image also reveals that along with the formed TiB_2 particles other intermediate particles have also formed. This shows that the reaction is not complete.

CONCLUSIONS

Overall, the prepared composites at different stirring medium had greater impact on the microstructural and mechanical properties than the unreinforced alloy, according to the findings. Several causes can be attributed to the properties of the composites:

Manual stirring of the melt with salts was found to be desirable condition for production Al1050-5%TiB_2 composite. Composite processed with motorized stirring of melt with mixture of K_2TiF_6 and KBF_4 salts has led to formation of more unwanted AlB_2 particles, Al_3Ti particles and Slag.Composite processed without stirring of the melt with the stoichiometry mixture of K_2TiF_6 and KBF_4 salts led to presence of more unwanted slag in the prepared composite.Motorized stirring of the melt led to the presence of unwanted slag and salts in the melt which in turn increased the viscosity of the melt which led to the difficulty in pouring of the composite meltThe composites' characterization shows that without stirring the slag in the matrix alloy also increased due to the uneven mixing of salts with the melt. As a result, the above-mentioned causes could be to blame for the composites' decreased qualities. Based on the knowledge gathered from this opinion, it was decided stirring the melt manually was desirable condition for obtaining the best results.The overall observations also confirmed that the Stirring medium also had an important role in deciding the properties of the prepared composite.

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